ADSORPTION OF CARBON DIOXIDE AND METHANE ON ALKALI METAL EXCHANGED SILICOALUMINOPHOSPHATE ZEOLITE

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To my dear parents, siblings and my late sister Amina Umaru Baba. Allah's Rahma on your soul our beloved sister. You will always be in our hearts.

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ABSTRACT

Aluminosilicate zeolites remain the most widely used microporous materials for important industrial gas separation applications. Aluminophosphate molecular sieves (AlPO-n) are the first microporous materials synthesized without silica. Substitution of silica in the AlPO-n framework result in the formation of silicoaluminophosphate (SAPO-n) molecular sieve. In this study, Na-SAPO-34 zeolite was synthesized and ion exchanged with alkali metal cations (K^+ and Li^+) using a two-step ion exchange procedure, with each step lasting for 24 hours. The prepared materials were characterized using X-ray diffraction (XRD), field emission scanning electron microscopy (FESEM), inductive coupled plasma optical emission spectroscopy (ICP-OES), and Fourier transform infrared spectroscopy (FTIR). Thermal analysis was carried out using thermographymetric analyzer (TGA) and differential scanning calorimetry (DSC). The textural properties (surface area, pore size and pore volume) were calculated from N_2 adsorption isotherm at 77 K. Equilibrium adsorption isotherms and adsorption capacity were measured volumetrically at 273, 298 and 323 K respectively for CO_2 and at 298 K for CH_4 at pressure of 101.3 KPa. The corresponding isosteric heats of adsorption of CO_2 was calculated using the Clausius-Claperon equation. The Langmuir, Freundlich and Sips models were applied to correlate the adsorption isotherm data. The equilibrium performance of the prepared materials for CO_2 separation from CO_2/ CH_4 mixtures operating at room temperature were calculated based on working capacity, regenerability and selectivity. The results showed that although all the prepared materials show changes in structural properties, the structure did not collapse after ion exchange. All the prepared materials showed high preference for CO_2 over CH_4. At 273 K, the 1st stage K^+ ion exchange material had the highest CO_2 adsorption capacity (2.90 mmol/g), whereas at 323 K, the 1st stage Li^+ material had the highest capacity (1.90 mmol/g). However, at 298 K, the adsorption capacity did not improve. The 1st stage K^+ material had highest adsorption capacity for CH_4 (0.54 mmol/g) at 298 K. Sips model provides the best fit with experimental data. The 1st stage K^+ ion exchange material had the highest heat of adsorption reflecting stronger interaction between the extra framework cation and CO_2. Based on the adsorbent performance evaluation, all the prepared materials showed potential for CO_2 removal from CO_2/ CH_4 mixtures at room temperature with the exception of the 2nd stage K^+ ion exchange material. The lithium ion exchange series had higher working capacity and regenerability than the potassium ion exchanged series at 298 K. This study showed that adsorption capacity of CO_2 and CH_4 depends not only on temperature but also on the properties of the exchangeable cation such as size, charge, location, and distribution within the channels and cavities of the prepared materials.

ABSTRAK

Zeolit aluminosilikat kekal menjadi bahan berliang mikro yang paling banyak digunakan untuk aplikasi penting dalam pemisahan gas perindustrian. Molekul saringan aluminofosfat (AlPO-n) adalah bahan berliang mikro pertama yang disintesis tanpa silika. Penggantian silika di dalam rangka AlPO-n bagi menghasilkan molekul saringan silikoaluminofosfat (SAPO-n). Dalam kajian ini, Na-SAPO-34 zeolit telah disintesis dan ion ditukar dengan kation logam alkali (K^+ dan Li^+) menggunakan prosedur penukaran ion dua-langkah, dengan setiap langkah selama 24 jam. Bahan yang disediakan telah dicirikan menggunakan pembelauan sinar-X (XRD), mikroskopi imbasan elektron pancaran medan (FESEM), spektrometer pancaran optik plasma berganding aruh (ICP-OES), dan spektroskopi Fourier inframerah transformasi (FTIR). Analisis terma dijalankan menggunakan penganalisis termogravimetri (TGA) dan kalorimeter imbasan perbezaan (DSC). Ciri-ciri tekstur (luas permukaan, saiz liang dan isipadu liang) telah daripada penyerapan isoterma N_2 pada 77 K. Keseimbangan penyerapan isoterma dan kapasiti penyerapan diukur secara volumetrik pada 273, 298 dan 323 K bagi CO_2 dan 298 K bagi CH_4, pada tekanan 101.3 kPa. Haba isosterik bagi penyerapan CO_2 yang sepadan telah dikira menggunakan persamaan Clausius-Clapeyron. Model Langmuir, Freundlich dan Sips untuk mengaitkan data penyerapan isoterma. Prestasi keseimbangan bagi bahan yang disediakan untuk pemisahan CO_2 dari campuran CO_2/CH_4 pada suhu bilik telah dikira berdasarkan kepada kapasiti kerja, kebolehjanaan semula, dan kepemilihan. Keputusan telah menunjukkan walaupun berlaku perubahan pada sifat-sifat struktur namun struktur bahan yang disediakan tidak runtuh selepas penukaran ion. Semua bahan yang disediakan telah menunjukkan kecenderungan yang tinggi terhadap CO_2 berbanding CH_4. Pada 273 K, bahan ion tukaran K^+ tahap pertama mempunyai kapasiti menyerapan CO_2 yang tertinggi (2.90 mmol/g), manakala pada 323 K, bahan ion tukaran Li^+ tahap pertama mempunyai kapasiti tertinggi (1.90 mmol/g). Walau bagaimanapun, pada 298 K, kapasiti penyerapan tidak meningkat. Bahan ion tukaran K^+ tahap pertama mempunyai kapasiti penyerapan CH_4 tertinggi (0.54 mmol/g) pada 298 K. Model Sips memberikan padanan terbaik dengan data eksperimen. Bahan ion tukaran K^+ tahap pertama mempunyai haba penyerapan tertinggi yang menggambarkan interaksi yang lebih kuat antara kation rangka tambahan dan CO_2. Berdasarkan penilaian prestasi bahan penyerap, semua bahan yang disediakan menunjukkan potensi bagi penyingkiran CO_2 dari campuran CO_2/CH_4 pada suhu bilik dengan pengecualian bagi bahan tukaran ion K^+ tahap kedua. Siri penukaran ion Li^+ mempunyai kapasiti kerja dan kebolehjanaan semula yang lebih tinggi berbanding siri penukaran ion kalium pada suhu 298 K. Kajian ini telah menunjukkan kapasiti penyerapan CO_2 dan CH_4 bukan hanya bergantung sunu tetapi kepada ciri-ciri kation tukaran seperti saiz, cas, lokasi, dan taburan di dalam saluran dan rongga bahan yang disediakan.

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LIST OF ABBREVIATIONS

Al	-	Aluminium
Ar	-	Argon
ALPO-n	-	Aluminophosphate
Al_2O_3	-	Aluminium oxide
$Al_2O_3.3H_2O$	-	Bauxite
BDDT	-	Brunauer, Deming, Deming and Teller
BET	-	Brunauer, Emmette and Teller
CH_4	-	Methane
C_2H_6	-	Ethane
C_3H_8	-	Propane
C_4H_{10}	-	Butane
CHA	-	Chabazite
CO_2	-	Carbon dioxide
Cs	-	Cesium
D-A	-	Dubinin and Astakhov
DEA	-	Diethanolamine
DSC	-	Differential scanning calorimetry
FESEM	-	Field emission scanning electron microscopy
Fr	-	Francium
FTIR	-	Fourier transform infrared spectroscopy
H_2S	-	Hydrogen sulfide
He	-	Helium
HK-CY	-	Horvath and Kawazoe-Cheng and Yang
HTlcs	-	Hydrotalcite-like compounds
ICP-OES	-	Inductive coupled plasma-optical emission spectroscopy
IR	-	Infrared

IUPAC	-	Internation Union of Pure and Appled Chemistry
K^+	-	Potassium cation
KBr	-	Potassium bromide
L^+	-	Lithium cation
LNG	-	Liquefied natural gas
M^+	-	Monovalent cation
M^{2+}	-	Divalent cation
M^{3+}	-	Trivalent cation
MDEA	-	Methyl diethanolamine
MOF	-	Metal organic framework
Na^+	-	Sodium cation
Na-SAPO-34		Sodium-silicoaluminophosphate-34 zeolite
N_2	-	Nitrogen
N_2O	-	Nitrous oxide
Ne	-	Neon
O_2	-	Oxygen
OH	-	Hydroxyl group
P_2O_5	-	Phophorous pentoxide
P/VSA	-	Pressure/vacuum swing adsorption
PSD	-	Pore size distribution
Rb	-	Rubium
SAPO	-	Silicoaluminophosphate
SBU	-	Secondary building units
SERP	-	Sorption enhanced reaction process
SiO_2	-	Silicon dioxide
Sr^{3+}	-	Strontium cation
TEA	-	Triethanolamine
TETA	-	Triethylenetetramine
TGA	-	Thermogravimetric analysis
TSA	-	Temperature swing adsorption
Tscf	-	Trillion standard cubic feet
Xe	-	Xenon

LIST OF SYMBOLS

$-\Phi$	-	Sorbate-sorbent interaction energy
$-\Delta G$	-	Free energy change
P	-	Pressure
P_0	-	Saturation pressure
P/P_0	-	Relative pressure
R	-	Universal gas constant
V	-	Physical volume of container
Φ_D	-	Dispersion energy
Φ_R	-	Repulsion energy
Φ_P	-	Polarization energy
$\Phi_{F\mu}$	-	Field-dipole interaction energy
Φ_{FQ}	-	Field-gradient quadruple interaction energy
Φ_{SP}	-	Adsorbate-adsorbate interaction energy
θ	-	Surface coverage
q	-	Quantity of gas adsorbed
q_m	-	Quantity of gas adsorbed when the entire surface is covered with a monolayer
C	-	BET constant
W	-	Quantity of gas adsorbed at relative pressure
W_0	-	Limiting micropore volume
β	-	Affinity constant
D_p	-	Particle size
B	-	Dimensionless factor equal to 0.94
λ	-	X-Ray wavelength
β	-	Line broadening at half the maximum intensity
θ_B	-	Bragg angle

K	-	Avagadro's number
N_a	-	Number of atoms per unit area of adsorbate
N_A	-	Number of moles per unit area of adsorbate
A_a	-	Constants in the Lennard-Jones potential for the adsorbent
A_A	-	Constant in the Lennard-Jones potential for the adsorbate
l	-	Distance between two nuclei of two layers
d	-	Diameter of the adsorbed molecule
V_0	-	Limiting micropore volume
n_{DA}	-	Dubinin-Astakhov parameter
n	-	Number of moles of a gas
V_m	-	Manifold volume
V_s	-	Reference volume
V_v	-	Void volume of sample
P_m	-	Manifold pressure
T_m	-	Manifold temperature
V_{vf}	-	Free space volume
V_d	-	Total volume of the adsorptive dosed
q_{st}	-	Isoteric heat of adsorption
b	-	Langmuir constant
q_s	-	Maximum amount adsorbed
H_K	-	Henry's constant
n_F	-	Heterogeniety parameter in the Freundlinch equation
n_s	-	Heterogeneity parameter in the Sips equation
y	-	Data point
y_{mean}	-	Average value of y data
df	-	Degree of freedom
ΔN_1	-	Working capacity
$R\ (\%)$	-	Regenerability
$\alpha_{1,2}$	-	Selectivity

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CHAPTER 1

INTRODUCTION

1.1 Research Background

Global warming is one of the main challenges faced by the world today. For the past few decades, a steady rise in global temperatures due to the increase in the concentration of greenhouse gases such as carbon dioxide (CO_2), nitrous oxide (N_2O), and sulphur hexafluoride (SF_6) in the atmosphere has been observed. Among the greenhouse gasses, CO_2 has been identified as a largest contributor to the greenhouse effect. The burning of fossil fuel for energy accounts for about 80% of global energy consumption and is by far the major contributor to greenhouse gas emission. Furthermore, Global energy demand shows a 1.6 % average annual growth over the period 2010-2020 (Komiyama *et al.*, 2005). This has therefore prompted the need for an efficient greenhouse gas mitigation method.

Petroleum hydrocarbon is the main source of fuel today and is widely used in the industry and for transportation in automobile. Natural gas which comprises mainly of methane (CH_4) is one of the most important hydrocarbon gases and is used for power in electricity generation and in vehicles as an automotive fuel. In its pure form, natural gas is clean burning and emits the least amount of harmful chemicals into the environment. The past decades has witnessed a marked increase in the production of natural gas and it is projected to be the fastest growing fossil fuel globally in the next two decades at 2.1% p.a. (Ruehl and Giljum, 2011). This is partly due to sharp increase in petroleum prices and the stringent environmental policies and regulations. The

increase in the demand for natural gas has prompted for alternative, more efficient and environmentally friendly means for its production.

In addition to methane, the natural gas from gas well is composed of a very complex mixture of different components such as carbon dioxide, sulphur, water, nitrogen, mercury, hydrocarbons (light, heavy and aromatic) and other impurities. Carbon dioxide removal from natural gas is of significant importance and must be removed to avoid formation of solids in cryogenic units and corrosion in steel pipes, and to avoid reduction in the heating value of natural gas. The average proportion in gas wells is between 0.5-10 vol % and can be as high as 70 vol % in some wells. Thus, the removal of CO_2 from crude natural gas during processing can be regarded as a key technology for the protection of the environment.

Adsorption is a unit operation used in the chemical, petrochemical and environmental industries mainly in applications involving bulk separation and purification of gases or the removal of trace impurities from gaseous mixture. It has become one of the most preferred gas separation technology and has attracted a lot of research interest especially in areas related to the environment.

Absorption using aqueous solutions of alkanolamine is among the most widely used methods for CO_2 removal from natural gas streams during processing (Rinker *et al.*, 2000). This method is only economically viable on a very large scale of operation. However, offshore gas fields require smaller and compact separation units that are also economical. Membrane separation (Baker, 2002; Komiyama, *et al.*, 2005), and cryogenic distillation (Baker and Lokhandwala, 2008) are among the technologies that have been explored in recent times. In spite the efficiency of these processes, their major drawbacks include the large amount of energy required for regeneration, equipment corrosion, solvent degradation in the presence of oxygen, high power requirement for refrigeration and membrane fouling. In view of the limitations of the conventional methods, separation of gases based on cyclic adsorption process such as pressure/vacuum swing adsorption (P/VSA) and temperature swing adsorption (TSA) using porous solid adsorbent materials is being proposed as an energy efficient

alternative mainly due to the lower energy requirement and operating costs and their applicability over a relatively wide temperature and pressures range of operation.

Aluminosilicate zeolites still remain the most widely used microporous adsorbent for important industrial applications. The continuous research into the design and synthesis of zeolites has resulted in the discovery of the aluminophosphate (ALPO) and silicoaluminophosphate (SAPOs) in the early 1980's. Their ability to reversibly take up molecules from liquid and gas phase is the basis for application in gas separation and purification. Silicoaluminophosphate (SAPOn) molecular sieves were discovered by the incorporation of Si into the framework of aluminophosphates (AlPO-*n*) molecular sieve (Deroche *et al.*, 2008). Phosphorus has been most successfully substituted by silicon. The direct replacement of isolated phosphorous atom by silicon results in a negatively charged framework. These charges are counter-balanced by exchangeable cations and, upon calcinations, an acid site (Bronsted acid sites). This can take the form of a bridging hydroxyl between aluminium and silicon atoms and are therefore similar in structure with those found in aluminosilicate zeolite but differ in acid properties. Similar to zeolites, the SAPOs are formed by channels and cavities of molecular dimensions. In fact, the same building units proposed in the formation of zeolites can be used to describe the SAPO frameworks. These materials have been reported to be exceptionally efficient in catalytic and adsorptive process (Claude and Martens, 2000; Dahl and Kolboe, 1994, 1996; Denayer *et al.*, 2004; Martens *et al.*, 1990)

1.2 Problem Statement

Absorption separation using liquid amine is one of the most widely used methods for CO_2 removal from natural gas, but it is only attractive economically on a very large scale operation. Thus, making this process only viable for very large gas processing fields. Consequently, new approaches have been proposed to overcome the limitations of the currently used liquid amine scrubbing technology which, in addition to its corrosive nature, consumes large amounts of energy during solvent regeneration.

Inspired by this technology, solid-supported amine adsorbents for CO_2 adsorption were developed, these materials are yet to be commercialized however, due to the energy penalty during regeneration resulting from strong chemical bonds formed from the reaction of amine with CO_2.

The adsorption property of zeolitic materials for any separation is very much dependent on the physiochemical properties of the surface. In spite of the success in sorbent design, there is still need for novel adsorbents that can be tailored with specific attributes to meet specific applications. In adsorption based separation process, the choice of the adsorbent is among the most crucial consideration. In literature, more attention was made mainly on ion exchange with the well-known aluminosilicate zeolites (Mace *et al.*, 2013; Sethia *et al.*, 2014; Walton *et al.*, 2006; Xu *et al.*, 2008). Although Na-SAPO-34 has shown great promise with regard to CO_2 adsorption and separation, it has not been fully utilized in gas separation application due to the lack of detailed characterization of its structure. Further structural studies are important in order to have more knowledge of the physiochemical properties of this material because the performance of any adsorption separation process is directly dependent upon the quality of the adsorbent. It is also vital to establish a relationship between the surface properties of SAPOs as adsorbents and their adsorption capacities.

Na-SAPO-34 is a sorbent with the chabazite (CHA) topology. The extra framework cations in Na-SAPO-34 can provide effective functionalization and affects its performance. A number of studies have been conducted on the effect of larger sized monovalent (Ag^{+}), divalent (Ca^{2+}, Mg^{2+}, Sr^{2+}) and trivalent (Ti^{3+}, and Ce^{3+}) ion exchanged Na-SAPO-34 zeolite for selective adsorption of nitrogen, methane and carbon dioxide (Arévalo-Hidalgo *et al.*, 2010; Rivera-Ramos and Hernández-Maldonado, 2007) at different temperatures. Adsorption capacity of the monovalent and divalent cation exchanged sorbents were found to increase after ion exchange only at 273K, while that of the trivalent cations decreased at all temperature. Similarly, the isoteric heat of adsorption indicated that the Sr^{+} ion exchanged sorbent had stronger interaction with CO_2 than the other sorbents including the unmodified one.

This research is conducted to study cation ability to access the framework of Na-SAPO-34 zeolite during ion exchange that may result in wider free volume, at the same time, produce higher electric field gradients which promote stronger interaction with the quadruple moment of CO_2. Potassium cation has been neglected due to its large size that can result in pore blockage. At present, no record has been found in literature dealing with CO_2 and CH_4 equilibrium adsorption on lithium and potassium ion exchange in Na-SAPO-34.

1.3 Aim and Objectives

The aim of this research is to synthesize some novel ion exchanged silicoaluminophosphate (Na-SAPO-34) zeolites using alkali metal cations via a two-step ion exchange procedure. The specific objectives include:

1. To synthesize and characterize Na-SAPO-34 zeolite and its modified samples.
2. To study the effect of ion exchange with K^+ and Li^+ on the adsorption properties of CO_2 at 273, 298, and 323 K and CH_4 at 298 K, at pressures up to 101.3 KPa.
3. To evaluate the potential of the prepared materials for the separation of CO_2 from its mixtures with CH_4.

1.4 Scope of Research

The scope of this research is based on the following:

1 Hydrothermal synthesis of Na-SAPO-34 zeolite and the subsequent ion exchange using Li and K cations

2 Structural and physiochemical Characterization of prepared materials using XRD, FESEM, FTIR, N_2 adsorption, ICP-OES, TGA and DSC and

3 Determining the potential of the synthesized materials for carbon dioxide adsorption at 273, 298 and 323 K, as well as the potential of the prepared materials for carbon dioxide separation from its mixture with methane at room temperature.

Ion exchanged was carried out in a two-step procedure using lithium and potassium cations. Characterization of the prepared materials was carried out using X-ray diffraction (XRD) analysis to determine the crystallinity of the prepared materials. Field Emission Scanning Electron Microscopy (FESEM) analysis to observe/visualize the topographical details and/or changes on the surface. Fourier Transform Spectroscopy (FTIR) analysis to identify the attachment of functional groups on the samples and their effect after ion exchange. N_2 adsorption studies was performed to determine the textural properties of the prepared materials before and after ion exchange. Elemental composition was determined using Inductive Coupled Plasma-Optical Emission Spectroscopy (ICP-EOS) technique, which provides a detailed concentration of elements present relative to a standard reference. The composition of each element was given as weight percentage of the solid weight. Thermal stability of the prepared materials were studied using Thermo-Gravimetric Analysis (TGA) to monitor the changes in weight of the sample as a function of temperature in a controlled atmosphere. Differential Scanning Calorimetriy (DSC) analysis was used to study the glass transition temperature, which measures the endothermic and exothermic heat flow of the prepared materials as a function of temperature/time. Adsorption isotherms and adsorption capacity were measure volumetrically and compared between the parent Na-SAPO-34 material and the ion exchanged samples.

1.5 Significance of Research

The continuous need to use natural gas in its pure form makes it paramount to investigate efficient and realistic means of separating it from other impurities. The development of technologies and/or materials that are capable of aiding this purification is very important. Cation exchange of micrporous adsorbent materials can significantly improve their CO_2 adsorption and selectivity.

The current study was carried out to investigate the use ion exchange as a means to modify the surface properties of Na-SAPO-34 zeolite using lithium and potassium as exchangeable cations and to further investigate the prepared materials for CO_2 adsorption and separation from its mixtures with CH_4. These sorbents provided some improvement in CO_2 adsorption at different temperatures as well as good selectivity from CO_2/CH_4, and could be applied in removing CO_2 from natural gas. The current study has never been carried out to date and thus represents the novelty of this research study.

1.6 Limitation

Although a lot of efforts has been put towards achieving the aim of this research, some factors have been unavoidably absent due to the limiting factors that may exist. Firstly, this study was conducted to investigate the prepared materials for the separation CO_2 from its mixtures with CH_4 at 298 K as such, the performance of the material in terms of separation were not investigated at other temperatures. Secondly, this research is limited to the thermodynamic studies and therefore, the kinetic studies was not included. Finally, structural characterization has been limited to the XRD, FTIR, FESEM, TGA, DSC and N_2 adsorption at 77 K. Although other novel and powerful characterization methods are required to better elucidate the structure of the materials, the lack of availability of the equipment has limited the characterization to the ones mentioned herein.

1.7 Report Outline

This report comprises five chapters. Chapter 1 introduces carbon dioxide and its effect on the environment, problems encountered in the industry due to the presence of CO_2 in natural gas stream and the underlining objectives to solve the problems. Chapter 2 contains a critical review on the aspects related to the field of research study. This includes natural gas and natural gas processing techniques, adsorption and gas separation techniques, membrane separation, adsorption isotherms and mechanism, microporous and mesoporous materials. The research methodology, comprising research materials, experimental procedures such as synthesis, ion exchange and detailed characterization and CO_2 sorption study are discussed in chapter 3. Results and discussion on the research findings is contained in chapter 4. Finally, chapter 5 contains the conclusions and recommendation for future work.

1.8 Summary

Adsorption has become a key separation tool in the chemical industry. Separation by adsorption is of great importance in industrial separation process and has witnessed tremendous growth in the past decades in areas such as air separation, hydrogen purification, carbon dioxide recovery and natural gas purification. Similarly, a lot of progress have been made in adsorbent design and process cycle development. The SAPO-34 zeolite have shown remarkable adsorption properties due to its unique channels and cavities which can be explored in applications involving adsorption and catalysis. The physiochemical properties of this material is very important during gas adsorption. Ion exchange is one of the promising methods used to modify the surface of zeolite materials in order to improve the adsorption characteristics without damaging the framework structure. An understanding of the physical and chemical structure of material after ion exchange is important in the design of novel adsorbents for use in adsorption process. Similarly, knowledge of the nature of adsorption and desorption phenomena is important in determining the mechanism of gas adsorption on the material and in identifying other valuable applications.

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